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# Nanomaterials by Severe Plastic Deformation

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# Properties of Aluminum Alloys Processed by Equal Channel Angular Pressing Using a 60 Degrees Die

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## 1 Abstract

Tests were undertaken to evaluate the significance of performing equal-channel angular pressing using a die having an angle of 60° between the two channels. The tests were conducted using samples of pure aluminum and an Al-1% Mg-0.2% Sc alloy. The results lead to the conclusion that the mechanical properties of samples in the as-pressed condition are similar for both 60° and 90° dies provided the data are compared at the same equivalent strains.

## 2 Introduction

Equal-channel angular pressing (ECAP) is a processing procedure in which severe plastic deformation is imposed on a sample by pressing it through a die contained within a channel that is bent through an abrupt angle [1]. It is now well-established that processing by ECAP has the potential for achieving very significant grain refinement in polycrystalline metals [2,3], generally to the submicrometer level and possibly even to the nanometer level. In practice, the strain imposed in a single passage through the die in ECAP depends upon two internal angles: the angle  $\Theta$  between the two parts of the channel and the angle  $\Psi$  representing the outer arc of curvature where the two channels intersect [4]. It can be shown that the angle  $\Theta$  is especially significant in determining the strain and in general the imposed strain is close to  $\sim 1$  for all values of  $\Psi$  when  $\Theta = 90^\circ$  [5].

Numerous experiments have been reported to date using dies having different values of  $\Theta$ . There are many reports where the value of  $\Theta$  is either 90° or 120° and there are also some reports of experiments using dies having larger angles up to 135°. To place these various results in perspective, a detailed evaluation of the as-pressed microstructures was conducted using ECAP dies having four different channel angles: specifically, the values of  $\Theta$  were 90°, 112.5°, 135° or 157.5°, respectively [6]. From these experiments it was concluded that an ultrafine microstructure of reasonably equiaxed grains, separated by boundaries having high angles of misorientation, is attained most readily when using a die having a channel angle that is close to, or equal to, 90° so that a very intense strain is imposed on each separate pass through the die. The implication from these results is that equiaxed microstructures may be achieved even more readily when the channel angle  $\Theta$  is reduced below 90° since this will give an even larger strain on each pass. However, there have been no reports to date of any experiments in which ECAP was conducted using a die having a value of  $\Theta < 90^\circ$ . Accordingly, the present experiments were initiated to evaluate the microstructures and mechanical properties which may be achieved in pure aluminum and an Al-based alloy when using a die having a channel angle of  $\Theta = 60^\circ$ .

### 3 Experimental Materials and Procedures

The experiments were conducted using aluminum of 99.99 % purity and an Al-1 wt % Mg-0.2 wt % Sc alloy. The pure Al was supplied in a hot-rolled condition and it was homogenized for 24 hours at 753 K, swaged into a rod with a diameter of 10 mm and then the rod was cut into lengths of ~60 mm. Prior to ECAP, the pure Al was annealed for 1 hour at 773 K and air cooled to give an initial grain size of ~1 mm. The Al-Mg-Sc alloy was prepared by casting into an ingot with dimensions of  $18 \times 60 \times 160 \text{ mm}^3$  and it was also homogenized for 24 hours at 753 K, cut into bars with dimensions of  $15 \times 15 \times 120 \text{ mm}^3$  and then swaged and cut to the same dimensions as for the pure Al. The alloy was given a solution heat treatment for 1 hour at 883 K followed by rapid quenching. The grain size of the alloy prior to ECAP was ~500  $\mu\text{m}$ . Further details on the ECAP processing of these materials when using a die with  $\Theta = 90^\circ$  were given earlier for pure Al [5,7] and the Al-1% Mg-0.2% Sc alloy [8], respectively. Information is also available on ECAP of an Al-3% Mg-0.2% Sc alloy [9-12].

All of the pressings were conducted using a solid ECAP die constructed from SKD11 tool steel (Fe-1.2~1.4wt% C-11~13% Cr-0.8~1.2% Mo-0.5% V). The die was fabricated with an internal angle of  $\Theta = 60^\circ$  and special steps were taken to determine the value of the internal angle representing the arc of curvature,  $\Psi$ . When the sample passes through the shearing plane at the intersection of the two channels, it is sheared into a rhombohedral shape [13] such that the outer dimensions at the sheared end of the sample provide a direct measure of the shear strain,  $\gamma$ . Thus, using the conventional relationship for the imposed strain in ECAP [4] and taking  $\Theta = 60^\circ$ , the imposed strain,  $\epsilon (= \gamma/\sqrt{3})$ , was estimated as ~1.6 corresponding to an internal arc of curvature of  $\Psi \approx 30^\circ$ . Thus, each pass through the die imposes a strain of ~1.6 and the total strain is therefore  $1.6N$  where  $N$  is the number of passes. All of the pressings were conducted at room temperature with pure Al taken to a maximum of 4 passes and the Al-Mg-Sc alloy to a maximum of 8 passes. All specimens were pressed using route  $B_C$  where the samples are rotated by  $90^\circ$  in the same sense between each pass [14] and it has been established that this route is preferable for attaining an equiaxed microstructure [15].

Following ECAP, samples were examined using optical microscopy (OM) and transmission electron microscopy (TEM). Selected area electron diffraction patterns were recorded using an aperture size of ~12.3  $\mu\text{m}$ . Tensile specimens were machined from the as-pressed rods with gauge lengths of 5 mm and cross-sectional areas of  $3 \times 2 \text{ mm}^2$ . Tensile tests were performed at 673 K and the specimens were heated to the testing temperature over a period of ~30 minutes and then held at temperature for ~10 minutes prior to the start of each test. Specimens were pulled to failure at strain rates from  $1.0 \cdot 10^{-3}$  to  $3.3 \cdot 10^{-2} \text{ s}^{-1}$  and air cooled.

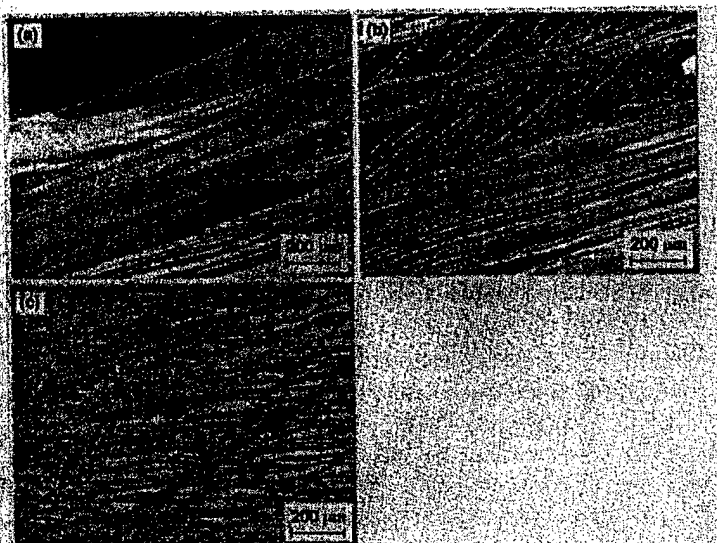
## 4 Experimental Results and Discussion

### 4.1 Microstructures after ECAP Using a $60^\circ$ Die

In order to understand the nature of the deformation occurring when a sample passes through an ECAP die having a channel angle of  $\Theta = 60^\circ$ , it is first necessary to construct the shearing patterns associated with this type of die. An earlier report documented the shearing patterns associated with dies having  $\Theta = 90^\circ$  and  $120^\circ$  [16] and the equivalent patterns for  $\Theta = 60^\circ$ , including

Route	Plane	Number of pressings					
		0	1	2	3	4	5
60° B <sub>C</sub>	X						
	Y						
	Z						

**Figure 1:** Schematic illustrations of grain shapes and shearing patterns on the X, Y and Z planes using a 60° die in route B<sub>C</sub>: results are shown for a total of 5 passes

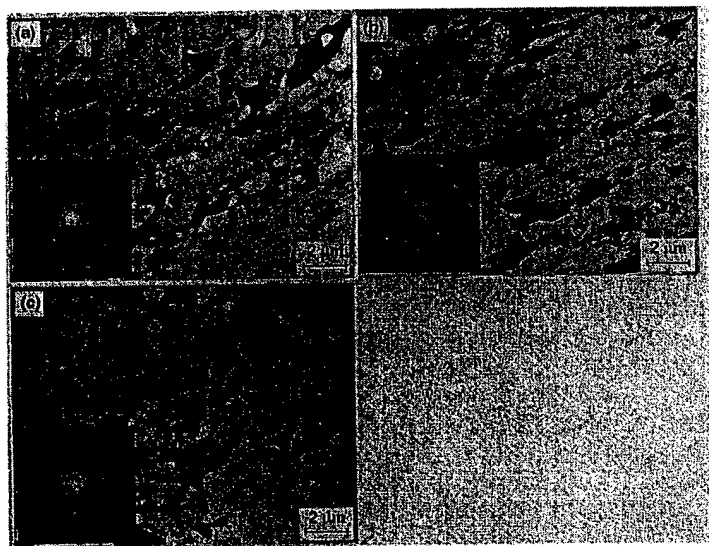


**Figure 2:** Optical microstructures of pure Al on the Y plane after ECAP using a 60° die and route B<sub>C</sub> for totals of (a) 1 pass, (b) 2 passes and (c) 4 passes

the grain distortions, are shown in Fig. 1 when using route B<sub>C</sub>: as previously, the X, Y and Z planes correspond to the transverse, flow and longitudinal planes, respectively.

Figures 2 and 3 provide microstructural information on the Y plane after ECAP for the samples of pure Al using OM and TEM, respectively: these specimens were pressed through the 60° die using route B<sub>C</sub> for totals of (a) 1 pass with a strain of ~1.6, (b) 2 passes with a strain of ~3.2 and (c) 4 passes with a strain of ~6.4.

Inspection of Figs 2(a) and (b) shows that the original grains are now inclined by ~16° to the X axis after 1 and 2 passes, and this is in agreement with the prediction given for the Y plane in



**Figure 3:** Microstructures of pure Al on the Y plane using TEM after ECAP with a 60° die and route B<sub>C</sub> for totals of (a) 1 pass, (b) 2 passes and (c) 4 passes

the central row of Fig. 1. After 4 passes shown in Fig. 2(c), there is an essentially equiaxed array of grains as also predicted for  $N = 4$  in Fig. 1. Thus, on the macroscopic scale, there is excellent agreement between the experimental observations and the theoretical predictions for a die with a channel angle of 60°. An earlier investigation demonstrated similar agreement between experiment and theory when using a conventional die with  $\Theta = 90^\circ$  [17]. For the TEM photomicrographs, Fig. 3 (a) shows bands of subgrains inclined at  $\sim 30^\circ$  to the X-axis and with a width of  $\sim 0.8 \mu\text{m}$  whereas in Fig. 3(b) the subgrains are inclined at  $\sim 15^\circ$ – $30^\circ$  from the X-axis: the SAED patterns after 1 and 2 passes are net patterns demonstrating the presence of low-angle boundaries although the diffraction spots tend to be slightly broader in Fig. 3(b). In Fig. 3(c) after 4 passes, the diffraction spots are distributed randomly and careful observations suggested  $\sim 70\%$  of the total area contained high-angle boundaries and  $\sim 30\%$  of the total area contained low-angle boundaries. In this condition, there is an array of essentially equiaxed grains with an average grain size of  $\sim 1.1 \mu\text{m}$  which is only marginally smaller than the grain size of  $\sim 1.2 \mu\text{m}$  recorded in pure Al with  $\Theta = 90^\circ$  [5,7]. These TEM observations are generally consistent with the predictions in Fig. 1.

#### 4.2 Mechanical Properties at Elevated Temperatures after ECAP Using a 60° Die

The preceding results show that the grain size attained in pure Al with a 60° die ( $\sim 1.1 \mu\text{m}$ ) is only slightly smaller than the grain size achieved in the same material with a 90° die ( $\sim 1.2 \mu\text{m}$ ). A similar result was found also for the Al-1% Mg-0.2% Sc alloy where the measured grain size on the Y plane after a total of 8 passes was  $\sim 0.31 \mu\text{m}$  which compares with  $\sim 0.36 \mu\text{m}$  when using a 90° die for the same number of passes [8,18]. It was shown earlier that these grains are reasonably stable at high temperatures [8,18] but nevertheless there was some grain growth in the period of  $\sim 40$  minutes required for heating to the test temperature and temperature stabiliza-

tion. For example, for the material pressed through a total of 8 passes with a 60° die the grain size was  $\sim 1.3 \mu\text{m}$  at the start of tensile testing whereas the grain size was  $\sim 1.2 \mu\text{m}$  in the material pressed through a total of 8 passes with a 90° die.

Figure 4(a) shows the variation of the elongation to failure with the imposed strain rate for specimens tested at 673 K using a range of strain rates. The results in Fig. 4(a) represent three different processing conditions: with a 90° die for 8 passes where the elongations are the lowest, with a 60° die for 6 passes where the elongations are intermediate and with a 60° die for 8 passes where the elongations are a maximum. At a strain rate of  $3.3 \cdot 10^{-3} \text{ s}^{-1}$ , the elongations to failure are 560 %, 800 % and 1020 % for these three pressing conditions, respectively. Thus, although the grain sizes in the two materials pressed through 8 passes are reasonably similar for the 60° and 90° dies, there is a significantly lower elongation to failure in the material pressed through the conventional 90° die. The reason for this apparent disparity can be seen by inspection of Fig. 4(b) where the elongation to failure with a strain rate of  $3.3 \cdot 10^{-3} \text{ s}^{-1}$  is plotted as a function of the equivalent strain for an unpressed sample, for samples pressed through a 60° die and for the sample pressed for 8 passes through a 90° die. Thus, the experimental point for the sample pressed through the 90° die lies at a similar equivalent strain, and shows a similar elongation to failure, as the sample pressed through only 5 passes with a 60° die. These results suggest that the fraction of high-angle boundaries probably increases with increasing equivalent strain in ECAP.

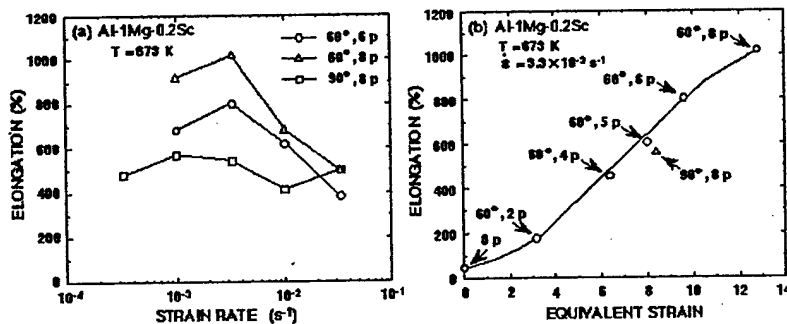


Figure 4: Elongation versus (a) strain rate and (b) equivalent strain for specimens of the Al-Mg-Sc alloy tested at 673 K after pressing with 60° or 90° dies

## 5 Summary and Conclusions

1. Samples of pure aluminum and an Al-1% Mg-0.2% Sc alloy were processed by ECAP using a 60° die. The resultant grain sizes were only marginally smaller than those attained using a conventional 90° die.
2. Experiments show the elongations to failure are significantly larger when processing with the 60° die but the elongations are essentially identical when specimens processed using 60° and 90° dies are compared at similar equivalent strains.

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